Influence of the built-in pyridinium salt on asymmetric epoxidation of substituted chromenes catalysed by chiral (pyrrolidine salen)Mn(III) complexes

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Abstract

Chiral (pyrrolidine salen)Mn(III) complexes **1** with an *N*-benzoyl group and **2** with an *N*-isonicotinoyl group as well as the corresponding *N*-methyl (**3**) and *N*-benzyl (**4**) pyridinium salts of **2** were synthesized. The catalytic properties of **1–4** and **2** with excess CH_3I were explored to figure out the influence of the internal pyridinium salt in the catalyst on asymmetric epoxidation of substituted chromenes with NaClO/PPNO as an oxidant system in the aqueous/organic biphasic medium. The (pyrrolidine salen)Mn(III) complexes with an internal pyridinium salt, either formed in situ or isolated, displayed higher activities than analogous complexes **1**, **2** and Jacobsen's catalyst in the aforementioned reaction, with comparable high yields and ee values. The acceleration of the reaction rate is attributed to the phase transfer capability of the built-in pyridinium salt of the (salen)Mn(III) catalyst. The effect of the internal pyridinium salt on the epoxidation of substituted chromenes is similar to that of the external pyridinium salts and ammonium halides.
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1. Introduction

Chiral epoxides are versatile intermediates that can be readily converted into a wide variety of enantiomerically pure molecules by means of regio- and stereo-selective ring opening reactions. Various methods have been developed for the preparation of chiral epoxides in the last two decades [1]. The asymmetric epoxidation of unfunctionalized alkenes catalysed by chiral (salen)Mn(III) complexes is one of the most effective methods for the preparation of chiral epoxides [2,3]. In particular, Jacobsen and co-workers developed a practical asymmetric epoxidation procedure using a two-phase system, with an aqueous phase containing a cheap commercial bleach (NaClO) and an organic phase composed of a solution of substrates and Jacobsen's catalyst or its analogues (Fig. 1) [4], which displayed high enantioselectivity for asymmetric epoxidation of conjugated *cis*-di-, tri- and tetrasubstituted prochiral alkenes [5]. In view of practical application, there has been a compelling interest in the development of catalytic epoxidation systems that can increase reaction activity in the NaClO aqueous/organic biphasic system. Usually a pyridine *N*-oxide derivative is added to the aforementioned catalytic system to stabilize the catalyst by ligation and to increase the epoxidation reaction rate by drawing the active oxidant, HOCl, into the organic phase without affecting enantioselectivity [6,7]. The addition of some phase transfer catalysts could accelerate the (salen)Mn(III)-catalysed epoxidation reactions [8]. Moreover, it was reported that the epoxidation activity could be enhanced in the two-phase reaction medium using (salen)Mn(III) catalysts with a built-in phase transfer capability, constructed by introducing tertiary amine unit(s) to the salen ligand [9–11].
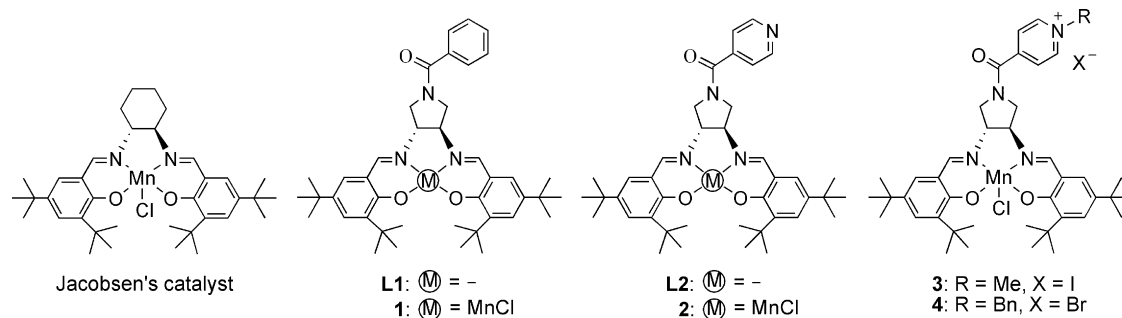
Fig. 1. Jacobsen's catalyst and chiral (pyrrolidine salen)Mn(III) complexes.

Very recently, we found that the addition of excess CH_3I to the biphasic system could increase the overall reaction rate of chromene epoxidation when (pyrrolidine salen)Mn(III) catalysts with a tertiary amine unit was used. The positive effect on the reaction rate is attributed to the formation of the quaternary ammonium unit in the molecule of the catalyst [12]. But this catalytic system has two drawbacks: (1) the enantioselectivity and the yield were slightly decreased in the presence of excess CH_3I, probably because the quaternary ammonium unit, formed on the N atom of the ligand and being close to the manganese center, might hinder the alkene's major access pathway and cause the decomposition of the catalyst; and (2) the (pyrrolidine salen)Mn(III) catalyst with a quaternary ammonium unit refused to be isolated from the reaction solution so that further studies on the catalysts with built-in phase transfer capability could not be continued. Taking into account aforementioned factors, in continuous studies on this catalytic system we designed and synthesized two chiral (pyrrolidine salen)Mn(III) complexes, one containing an *N*-benzoyl group (**1**) and the other featuring an *N*-isonicotinoyl group (**2**, Fig. 1). The pyridyl unit in complex **2** is far away from the manganese center and it can readily react with an alkyl halide to form a built-in pyridinium salt. Furthermore, complexes **3** and **4** with an internal pyridinium salt were successfully isolated from the reaction of complex **2** with iodomethane and benzyl bromide, respectively. The immediate aim of the present study is to explore the influence of the built-in pyridinium salt, either formed in situ or isolated from the reaction, on the asymmetric epoxidation of substituted chromenes, by comparing the catalytic activity and enantioselectivity of complexes **1–4** in the NaClO/PPNO biphasic system and by making an analogy between the effects of internal and external pyridinium salts and ammonium halides on the biphasic epoxidation reaction.

2. Experimental

2.1. Materials and instruments

4-Phenylpyridine *N*-oxide (PPNO) was purchased from Aldrich. Other commercially available materials were laboratory-grade reagents from local suppliers. *N*-Methylpyridinium iodide (*N*-MePyI) [13] and *N*-benzylpyridinium bromide (*N*-BnPyBr) were prepared by reported procedures [14]. 6-Nitro-2,2-dimethylchromene and 6-cyano-2,2-dimethylchro-

mene were synthesized according to the literature procedures [15]. Chiral ligand (3*R*,4*R*)-*N*-benzoyl-*N*′,*N*″-bis(3,5-di-*tert*-butylsalicylidene)-3,4-diaminopyrrolidine (H$_2$**L1**) and (3*R*,4*R*)-*N*-(isonicotinoyl)-*N*′,*N*″-bis(3,5-di-*tert*-butylsalicylidene)-3,4-diaminopyrrolidine (H$_2$**L2**) were prepared by following literature procedures [16–19]. Complex **1** was synthesized according to the previous report [19]. All solvents used were purified by standard procedures.

IR spectra were recorded from KBr pellets on a JASCO FT/IR 430 spectrophotometer and UV–vis spectra on an HP 8453 diode-array spectrophotometer. Mass spectra were performed by HR-ESI-MS on an HPLC-Q-TOF MS (micromass) mass spectrometer. The ee values of the epoxides of substituted 2,2-dimethylchromenes were determined by gas chromatography on a 6890N gas chromatograph (Agilent Co.) using a chiral capillary column (HP 19091G-B233, 30 m × 251 μm × 0.25 μm).

2.2. Synthesis of (pyrrolidine salen)Mn(III) complexes 2–4

(Pyrrolidine salen)Mn(III) complex **2** with an *N*-isonicotinoyl group was prepared according to the literature protocol with some modifications [17,20]. (Pyrrolidine salen)Mn(III) complexes **3** and **4** with a pyridinium ion were synthesized as follows:

A solution of complex **2** (0.36 g, 0.5 mmol) and CH_3I (0.16 mL, 2.5 mmol) or benzyl bromide (0.30 mL, 2.5 mmol) in CH_3CN (15 mL) was stirred at room temperature for 24 h and then concentrated under vacuum. The residue was purified by chromatography on a silica gel column ($MeOH/CH_2Cl_2$, 1:10) to give the desired complexes.

2: Yield 86%. IR (KBr): ν 2957, 2870, 1623, 1535, 1462 and 1433 cm^{-1}. UV–vis (CH_3OH): λ_{max} 290 (20,353), 322 (11,986), 426 (5210 mol^{-1} cm^{-1}) nm. HR-MS (ESI): *m/z* calcd for $[M - Cl]^+$ ($C_{40}H_{52}MnN_4O_3$): 691.3420, found: 691.3426.

3: Yield 0.23 g (53%). IR (KBr): ν 2955, 2873, 1620, 1535, 1462 and 1434 cm^{-1}. UV–vis (CH_3OH): λ_{max} 292 (18,192), 324 (11,027), 427 (4277 mol^{-1} cm^{-1}) nm. HR-MS (ESI): *m/z* calcd for $[M - Cl - I]^{2+}$ ($C_{41}H_{55}MnN_4O_3$): 353.1830, found: 353.1830.

4: Yield 0.40 g (89%). IR (KBr): ν 2956, 2869, 1622, 1536, 1456 and 1434 cm^{-1}. UV–vis (CH_3OH): λ_{max} 291 (21,788), 328 (13,480), 424 (6269 mol^{-1} cm^{-1}) nm. HR-MS (ESI): *m/z* calcd for $[M - Cl - Br]^{2+}$ ($C_{47}H_{59}MnN_4O_3$): 391.1984, found: 391.1979.

2.3. General procedures for asymmetric epoxidation of substituted chromenes using NaClO as oxidant [21]

A precooled NaClO aqueous solution (0.8 mmol, pH 11.3, 0 °C) was added portion-wise to a cooled solution (0 °C) of alkene (0.4 mmol), PPNO (13.7 mg, 0.08 mmol), *o*-dichlorobenzene (internal standard, 56 μL, 0.5 mmol), (pyrrolidine salen)Mn(III) complex (0.008 mmol) and 2 mol% of external pyridinium or *n*-tetrabutylammonium salt (if needed) in CH₂Cl₂ (1 mL). The mixture was stirred at 0 °C and the reaction was monitored by gas chromatography. When the conversion was steady, the mixture was diluted with CH₂Cl₂ (3 mL). The phases were separated and the aqueous layer was extracted with CH₂Cl₂ (3 mL × 2). The combined organic layer was washed with brine (3 mL × 2) and dried over anhydrous sodium sulfate. The concentrated filtrate was purified by chromatography on a silica gel column (EtOAc/petroleum ether, 1:5) to afford the corresponding epoxide.

2.4. Preparation of the pyridinium salt in situ and catalytic reaction by 2 and CH₃I

A solution of PPNO (13.7 mg, 0.08 mmol), a certain amount of CH₃I and (pyrrolidine salen)Mn(III) complex (0.008 mmol) in CH₂Cl₂ (1 mL) was stirred at room temperature for 1 h, and then alkene (0.4 mmol) and *o*-dichlorobenzene (internal standard, 56 μL, 0.5 mmol) were added to the aforementioned solution, and the mixture was cooled to 0 °C. The further catalytic reaction and the purification of epoxides were done as described in Section 2.3.

3. Results and discussion

3.1. Asymmetric epoxidation of substituted chromenes catalysed by 1 and 2

The catalytic activity and enantioselectivity of (pyrrolidine salen)Mn(III) complexes **1** and **2** were examined for asymmetric epoxidation of substituted chromenes using NaClO/PPNO as the oxidant system in CH₂Cl₂ at 0 °C (Scheme 1). 6-Nitro-2,2-dimethylchromene and 6-cyano-2,2-dimethylchromene were selected as representative substrates because their corresponding epoxides were important, biologically active compounds [22].
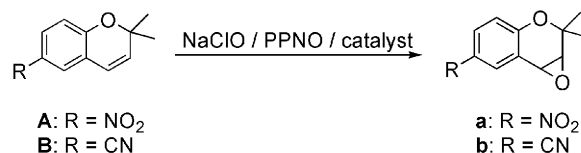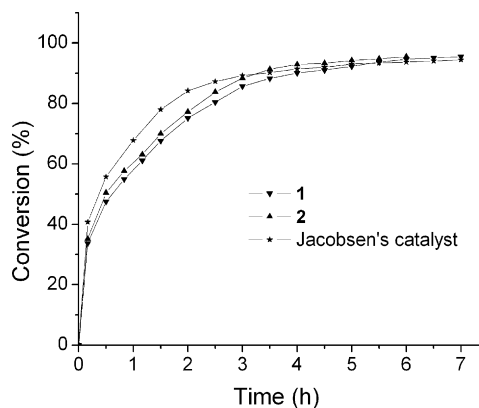

Scheme 1.

Fig. 2. The conversion vs. reaction time plot for epoxidation of 6-nitro-2,2-dimethylchromene catalysed by **1**, **2** and Jacobsen's catalyst with NaClO/PPNO as an oxidant system at 0 °C.

The catalytic results are summarized in Table 1. For comparison, catalytic results of the Jacobsen's catalyst under the same reaction conditions are also listed in Table 1. All reactions proceeded smoothly, and complexes **1** and **2** gave high yields and good ee values of the epoxides.

As shown in Fig. 2 and Table 1, under the same reaction conditions, complex **2** with an *N*-isonicotinoyl group showed activity, yield and ee value for epoxidation of substituted chromenes in the NaClO/PPNO aqueous/organic biphasic system similar to its analogous complex **1** with an *N*-benzoyl group, which indicates the introduction of a pyridyl group to the backbone of the catalyst shows no observable effect on the catalytic properties (entries 2 versus 1 and 5 versus 4). The catalytic results also revealed that (pyrrolidine salen)Mn(III) complexes **1** and **2** displayed slightly lower conversions than the Jacobsen's catalyst in the first 2 h. After 6 h, complexes **1** and **2** gave conversions, yields and ee values comparable to Jacobsen's catalyst (entries 1–6).

Table 1
Asymmetric epoxidation of substituted chromenes catalysed by complexes **1** and **2**[a]

Entry	Substrate	Catalyst	Time (h)	Yield (%)[b]	ee (%)[c]	Config.
1	A	**1**	6	94	91	3*R*, 4*R*
2		**2**	6	94	90	3*R*, 4*R*
3		Jacobsen's catalyst	6	92	90	3*R*, 4*R*
4	B	**1**	6	94	92	3*R*, 4*R*
5		**2**	6	96	91	3*R*, 4*R*
6		Jacobsen's catalyst	6	95	93	3*R*, 4*R*

[a] Reactions were carried out at 0 °C in CH₂Cl₂ (1 mL) with alkene (0.4 mmol), catalyst (0.008 mmol, 2 mol%), NaClO aqueous solution (pH 11.3, 0.8 mmol), PPNO (0.08 mmol) and *o*-dichlorobenzene (internal standard, 0.5 mmol).

[b] Isolated yields.

[c] Determined by GC with a chiral capillary column (HP19091G-B233, 30 m × 251 μm × 0.25 μm).

Table 2
Asymmetric epoxidation of 6-nitro-2,2-dimethylchromene catalysed by complexes **1** and **2** in the presence of CH₃I[a]
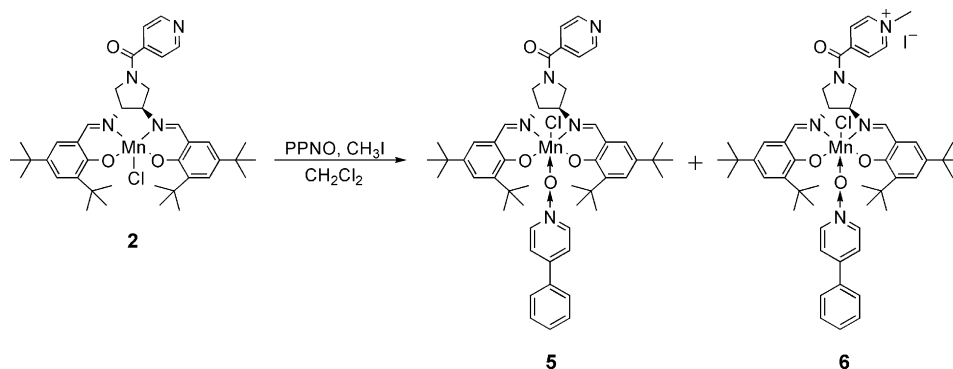
Entry	Catalyst	CH₃I (equiv.)[b]	Conversion (%) after 10 min	Time (h)[c] (conversion (%))[d]	ee (%)[e]	Config.
1	**1**		36	7 (96)	91	3R, 4R
7	**1**	50	38	7 (97)	92	3R, 4R
2	**2**		42	6 (95)	90	3R, 4R
8	**2**	20	55	4 (96)	90	3R, 4R
9	**2**	50	68	2.5 (96)	91	3R, 4R

[a] The reaction condition is the same as that in footnote 'a' of Table 1.
[b] Based on the mol of catalyst.
[c] The time needed to the end of the reaction.
[d] Determined by GC, no detectable amount of by-product was found.
[e] The same as that in footnote 'c' of Table 1.

3.2. Effect of the pyridinium salt formed in situ by complex 2 and CH₃I

The epoxidation of chromenes catalysed by chiral (salen) Mn(III) complexes with NaClO as oxidant under biphasic reaction conditions generally requires a long reaction time even in the presence of an axial ligand [22–24]. In order to shorten the reaction time, we studied the effect of the pyridinium salt formed in situ by complex **2** and CH₃I on asymmetric epoxidation of 6-nitro-2,2-dimethylchromene in the NaClO/PPNO aqueous/organic biphasic system. The catalytic results are presented in Table 2. The epoxidation of 6-nitro-2,2-dimethylchromene by **2** in the absence of CH₃I took 6 h to the end of the reaction (entry 2), whereas in the presence of 50 equiv. of CH₃I versus catalyst, the same epoxidation reaction was completed within 2.5 h in the identically high conversion and good ee value (entry 9). An increase in the amount of added CH₃I from 0 to 50 equiv. had an apparent positive effect on the overall reaction rate, especially in the beginning of the reaction (see Supplementary data, Fig. S1). For example, complex **2** gave a 42% conversion of 6-nitro-2,2-dimethylchromene in the absence of CH₃I in the first 10 min of the reaction (entry 2), while 55 and 68% conversions were obtained, respectively, in the presence of 20 and 50 equiv. of CH₃I versus catalyst in the same reaction period (entries 8 and 9). In contrast, complex **1** containing an *N*-benzoyl group with addition of CH₃I, even to 50 equiv. versus **1**, did not show considerable effect on the overall reaction rate, the conversion and the ee value in the epoxidation of 6-nitro-2,2-dimethylchromene (entries 1 versus 7).

To figure out the Mn(III) species formed in the presence of PPNO and CH₃I, the mixture of complex **2**, 10 equiv. of PPNO and 20 equiv. of CH₃I was stirred at room temperature for 1 h in CH₂Cl₂ and the solution was characterized by ESI-MS (see Supplementary data, Fig. S2). The MS spectrum of **2** with a pyridyl group showed that there existed two manganese-containing species, a PPNO axially-coordinated Mn(III) complex **5** ([$M - Cl$]⁺, found: $m/z = 862.3$, calcd: 862.4) and a corresponding pyridinium salt **6** ([$M - Cl - I$]²⁺, found: $m/z = 438.7$, calcd: 438.7) (Scheme 2). According to the experimental results, the enhancement of the overall reaction rate for epoxidation of 6-nitro-2,2-dimethylchromene by **2** in the presence of excess CH₃I is attributed to the formation of the pyridinium salt in the salen ligand of the catalyst, which possesses a built-in phase transfer capability and may facilitate the reaction in an aqueous/organic biphasic medium as reported for built-in ammonium salts [25]. In the previous studies we found that the time needed to the end of the reaction for the (pyrrolidine salen)Mn(III) complex featuring an *N*-benzyl group could be shortened from 6 to 2 h with the increase in the amount of CH₃I from 0 to 50 equiv., but that the conversion and enantioselectivity were somewhat decreased [12]. In contrast to the (pyrrolidine salen)Mn(III) complex with an *N*-benzyl group, complex **2** with an *N*-isonicotinoyl group displayed similar high conversions and good ee values in the absence or presence of CH₃I, probably because the pyridinium ion formed in situ is far away from the manganese center so that it could not cause the decomposition of the catalyst and could not influence the alkene's major access pathway.

Scheme 2.

Table 3
Asymmetric epoxidation of substituted chromenes catalysed by complexes **3** and **4**[a]

Entry	Substrate	Catalyst	Conversion (%) after 10 min	Time (h)[b] (conversion (%))[c]	ee (%)[d]	Config.
2	A	**2**	42	6 (95)	90	3R, 4R
10		**3**	72	1.5 (93)	86	3R, 4R
11		**4**	66	2 (94)	87	3R, 4R
5	B	**2**	–	5.5 (96)	91	3R, 4R
12		**3**	–	1.5 (94)	88	3R, 4R
13		**4**	–	2 (93)	88	3R, 4R

[a] The reaction condition is the same as that in footnote 'a' of Table 1.
[b] The time needed to the end of the reaction.
[c] Determined by GC, no detectable amount of by-product was found.
[d] The same as that in footnote 'c' of Table 1.

3.3. Effect of the built-in pyridinium salt on asymmetric epoxidation of substituted chromenes catalysed by 3 and 4

To further study the aforementioned built-in phase transfer capability, we tried to isolate the intramolecular pyridinium salts of the (pyrrolidine salen)Mn(III) complex **2**, i.e. **3** and **4**. The catalytic performance of **3** and **4** in asymmetric epoxidation of substituted chromenes were examined in the NaClO/PPNO biphasic system. As shown in Fig. 3 and Table 3, under the identical reaction conditions, the reaction rates for complexes **3** and **4** with a pyridinium ion were similar to that for **2** with 50 equiv. of CH$_3$I for epoxidation of 6-nitro-2,2-dimethylchromene (entries 9 versus 10, 11), and the time required to the end of the reaction was significantly shortened as compared to that for **2** with a pyridyl group for epoxidation of nitro- and cyano-substituted 2,2-dimethylchromenes (entries 2 versus 10, 11 and 5 versus 12, 13). The overall reaction rate of epoxidation of substituted chromenes, especially in the beginning of the reaction, was considerably enhanced by using complexes **3** and **4** featuring a built-in pyridinium ion. For example, complex **2** gave a 42% conversion of 6-nitro-2,2-dimethylchromene in the first 10 min of the reaction (entry 2), while 72 and 66% conversions were obtained, respectively, for complexes **3** and **4** in the same reaction period (entries 10 and 11). Comparable conversions and slightly decreased ee values were obtained with **3** and **4** as catalysts.
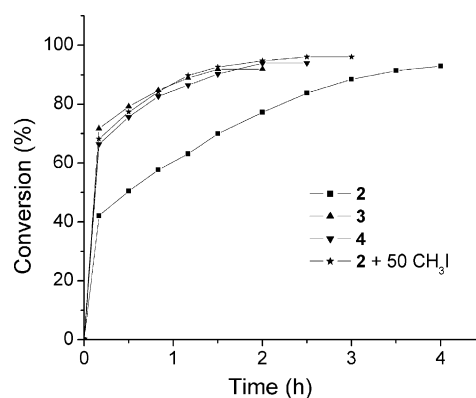
Table 4
Asymmetric epoxidation of 6-nitro-2,2-dimethylchromene catalysed by **2** in the presence of various pyridinium and tetrabutylammonium salts

Entry	Salt[a]	Time (h)[b]	Yield (%)[c]	ee (%)[d]	Config.
14	*N*-MePyI	2.5	94	89	3R, 4R
15	*N*-BnPyBr	3	95	90	3R, 4R
16	*n*-Bu$_4$NBr	2	95	90	3R, 4R
17	*n*-Bu$_4$NCl	3	96	88	3R, 4R
18	*n*-Bu$_4$NI	2	96	89	3R, 4R
19	*n*-Bu$_4$NHSO$_4$	15	79	87	3R, 4R
20	*n*-Bu$_4$NH$_2$PO$_4$	15	67	88	3R, 4R

[a] Pyridinium or tetrabutylammonium salt (2 mol%) was added. The other reaction condition is the same as that in footnote 'a' of Table 1.
[b] The time needed to the end of the reaction.
[c] Isolated yield.
[d] The same as that in footnote 'c' of Table 1.

3.4. Effect of external pyridinium and ammonium salts on asymmetric epoxidation of substituted chromenes catalysed by 2

In order to compare the effects of the built-in pyridinium salts and the external pyridinium and ammonium salts on the catalytic epoxidation by complex **2**, we also studied the epoxidation of 6-nitro-2,2-dimethylchromene in the NaClO/PPNO biphasic system in the presence of external pyridinium and *n*-tetrabutylammonium salts. The results are listed in Table 4. The epoxidation of 6-nitro-2,2-dimethylchromene catalysed by **2** took 6 h to the end of the reaction (entry 2), while in the presence of 2 mol% of pyridinium salts or *n*-tetrabutylammonium halides the same epoxidation reaction was completed within 2–3 h in high yields and good ee values (entries 14–18). The addition of 2 mol% of *n*-Bu$_4$NHSO$_4$ and *n*-Bu$_4$NH$_2$PO$_4$ greatly slowed down the epoxidation reaction, giving obviously lower yields and slightly decreased ee values (entries 19 and 20).

4. Conclusion

A chiral (pyrrolidine salen)Mn(III) complex **2** containing an *N*-isonicotinoyl group was prepared, which gave high yields and enantioselectivity in the asymmetric epoxidation of substituted chromenes with NaClO/PPNO as an oxidant system similar to Jacobsen's catalyst and the analogous complex **1** with an

Fig. 3. The conversion vs. reaction time plot for epoxidation of 6-nitro-2,2-dimethylchromene catalysed by **2–4** and **2** +50 equiv. of CH$_3$I with NaClO/PPNO as an oxidant system at 0 °C.

N-benzoyl group. The addition of excess CH$_3$I to the biphasic system could considerably facilitate the epoxidation reaction of 6-nitro-2,2-dimethylchromene catalysed by **2** with the high conversion and good enantioselectivity maintained, presumably due to the in situ formation of the internal pyridinium salt with built-in phase transfer capability. In contrast, the addition of excess CH$_3$I did not display considerable influence on the epoxidation of chromenes catalysed by complex **1**, which has neither a tertiary amine nor a pyridyl unit. As expected, the isolated complexes **3** and **4** featuring an internal pyridinium unit exhibited obviously higher activities than complexes **1** and **2** with comparable conversions and slightly decreased ee values, indicating that the internal pyridinium salt of the catalyst could play a built-in phase transfer function to greatly accelerate the epoxidation in aqueous/organic biphasic medium as 2 mol% of external pyridinium salts and ammonium halides.

Acknowledgements

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molcata.2007.01.052.

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